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QUALITY ASSURANCE MANUAL

Revised January 1984

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1. Introduction

This manual describes the Quality Assurance Program in use at Canton Analytical Laboratory, Inc., Ypsilanti, Michigan. It is intended for review by our clients and potential clients and internally as a reference for company personnel.

The information herein contained is the confidential property of Canton Analytical Laboratory, Inc. We ask that no copies of this document be made without the written consent of Canton Analytical Laboratory, Inc.

2. Purpose and Scope

The purpose of quality control is to monitor and control the generation of data. This program ensures that correct methodology, proper calibration and systematic quality control procedures are used. This manual describes in detail the analytical methods, laboratory services, instrumentation, glassware, reagents, analytical performance, sample handling, data processing and reporting, safety procedures, etc. which are all part of a comprehensive quality control program.

3. Analytical Methods

Most of the analytical methods used by Canton Analytical Laboratory have been documented by the Environmental Protection Agency (EPA) as approved methodologies.

Other methods are used for special projects.

An extensive list of sources employed may be found in the Reference Section.

Methodologies used for specific analyses are documented in Table I.

The methods which are routinely used, are contained in a Canton Analytical Laboratory Approved Methods Manual. As new methods attain acceptance, they are reviewed, changed as necessary, and become part of the "approved methods". The manual is reviewed and upgraded on an annual basis.

4. Sample Handling, Data Processing and Reporting of Results

Each sample entering the laboratory is assigned a number. This number is affixed to the sample container and recorded into a sample log book. The sample number, client, client sample identification, description of sample, date received, date report requested, parameters requested, and all necessary information about the samples are entered in the log book. A laboratory data sheet is completed for each group of samples received and accompanies the samples to the laboratory. A laboratory data sheet is shown in figure 1.

The sample is split and preserved as required (see Table II for details). Work order sheets are prepared by recording the sample number in the left hand column and placing a diagonal slash under the appropriate parameter listings. Examples of work order sheets are shown in Figures 2A - 2E. Samples with short holding times are delivered immediately to the designated analyst. All samples requiring refrigeration are stored at 40C until testing is completed; any remaining sample is removed to ambient storage and retained for a minimum of thirty days.

Analysts record their work in permanent record books. There is a book provided for each parameter tested by the laboratory.

CANTON ANALYTICAL LABORATORY

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4. Sample Handling, Data Processing and Reporting of Results Continued

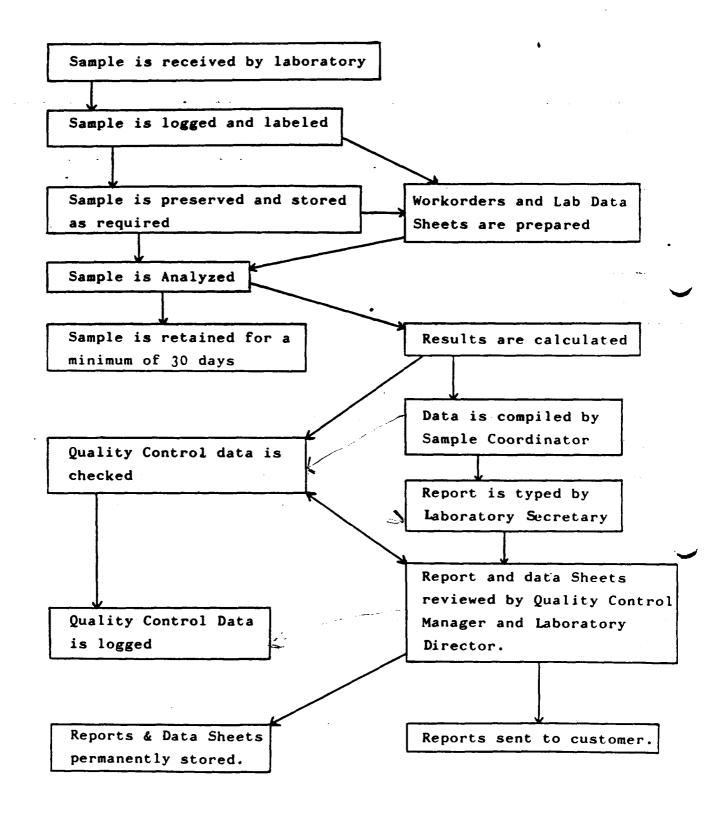
As the requested testing is completed, the analytical results are compiled on the laboratory data sheets and the work order forms are completed by marking an X in the appropriate parameter listings, indicating completion.

Completed laboratory data sheets are routed to the Sample Coordiator. The report is then reviewed by the Quality Assurance Coordinator for calculation errors, proper significant figures, and detection limits. Performance of quality control is verified as described in Sections 6 and 7.

The report is then typed and reviewed by the Quality Assurance Coordinator. The report is forwarded to the Laboratory Director for approval before being sent to the client.

The flow of work in the laboratory is shown in Figure 3.

CANTON ANALYTICAL LABORATORY SAMPLE FLOW CHART



5. Sample Bottles, Preservation and Holding Times

Canton Analytical Laboratory makes available to its clients both sample bottles and preservatives. A sample container requisition form is shown in Figure 4.

Canton Analytical Laboratory follows preservation, bottle and holding time guidelines described in EPA

Technical Addition to Methods for Chemical Analysis
of Water and Wastes, EPA-600/4-82-055, Dec. 1982, with
the exception of using glass bottles only for phosphate
where possible. Standard Methods for the Examination of
Water and Wastewater, 15th Ed., pg. 412 states, "Do not
store samples containing low concentrations of phosphorus
in plastic bottles unless kept in a frozen state because
phosphate may be absorbed onto the walls of plastic
bottles." The method of preservation along with the type
of bottle, volume of sample required and holding time for
each parameter is given in Table II.

SAMPLE CONTAINER REQUEST FORM

	Client:	_	Parameter:		
	Date:	•		•	
	Date Req. By:				
	Ship: UPS Other	·			
X	Deliver:				
•	Pick Up:				•
	.iumber of Sites:	_			
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	Final Disposition:	Deliver		Employee:	
		Pick-Up		Date:	
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Figure 4
Sample Container Request
Form

6. Chain of Custody Procedure

A. Introduction

The protocol developed by the USEPA (Appendix A) has been used as the framework for developing this procedure. It's purpose is to provide an accurate written record which can be used to trace the possession and handling of the sample(s) from collection through analysis and introduction as evidence.

A sample is in someone's "custody" if:

- It is in one's actual physical possession, or
- It is in one's view, after being in one's physical possession, or
- It is in one's physical possession and then locked up so that no one can tamper with it, or
- 4. It is kept in a secured area restricted to authorized personnel only.
- B. Sample Collection, Handling, and Identification
 A minimum number of people will be involved in sample collection and handling. In all instances, this will include the sample collector(s), the sample custodian, and the analyst(s).

6. Chain of Custody Procedure Continued

B. Sample Collection, Handling, and Identification Continued

A Field Sampling Report and Chain of Custody Form (Figure 5) will be complete at the time of sample collection by the sample collector(s).

All records and labels (Figure 6) will be filled out legibly in ink.

C. Transfer of Custody

Samples and records will be kept in "custody" as described above. When transferring the possession of the sample(s), the transferee must sign and record the data and time on the Chain of Custody Record. Every person who takes custody must fill in the appropriate section of the Chain of Custody Record.

All samples sent to the laboratory must be accompanied by the Chain of Custody Record and other pertinent forms. A copy of the forms should be retained by the originator (either carbon or photocopy).

Shipping receipts are retained as part of the permanent chain of custody documentation.

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CANTON ANALYTICAL LABORATORY: 153 Lider Street - Ypsilanti; MI 48197 Phone 313 483-7430 -

FIELD SAMPLING REPORT & CHAIN OF CUSTODY RECORD

Customer:					······································		
Plant Loca	ation:				·		
Site Descr	ription:						
Date:						·	
Sample .	Quantity	- [-	1	ime	By	Comm	nents
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ISIS OF FOOD AND WATER -

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CANTON ANALYTICAL LABORATORY

Environmental Analysis 153 Elder Street / Ypsilanti, MI 48197

(313) 483-7430

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6. Chain of Custody Procedure Continued

D. Laboratory Custody Procedure

Carol Sanford is the designated COC Custodian and Sandi Sibbitt is the alternate designated to act as custodian in the custodian's absence. All incoming samples are received by the custodian who indicates receipt by signing the accompanying custody forms and retains the signed forms as permanent records.

Permanent log books are maintained by the custodian for each and every sample.

All chain of custody records and samples are kept under lock and the keys are kept by the COC custodian. The custodian shall see that samples are properly stored and maintained prior to analysis.

Distribution of samples to the analysts will be made only by the custodian. To remove samples from storage, the analyst must sign for the samples on the chain of custody log sheet (Figure 7). The sample log sheet will be maintained by the custodian. Chain of custody samples must be returned to the custodian and to secured storage at the end of each day or when analyses are completed.

CHAIN OF CUSTODY SAMPLE LOG SHEET

Project	Sample	Signature of Analyst Responsible for Samples		Time		
Number	Numbers	Responsible for Samples	Date	Out	In	
						
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Figure 7 Chain of	Custody					
Sample I	.og					

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6. Chain of Custody Procedure Continued

D. <u>Laboratory Custody Procedure Continued</u>

Analysts must maintain sample custody as described in the introduction.

All analytical work is recorded in chain of custody log books. The books are distributed to the analyst(s) by the custodian. The log books must be returned to the custodian and to the secured file at the end of each day or when analyses are completed. The unused portion of samples will be retained under "custody" until destroyed or returned to the client. All laboratory records will be retained in the locked chain of custody file.

E. <u>Laboratory Security</u>

All laboratory doors will be kept locked to assure access to laboratory employees only or non-employees accompanied by a laboratory employee.

7. Monitoring Analytical Performance

All routinely performed tests are subject to initial evaluation of precision and accuracy. In addition, daily monitoring of precision and accuracy is done to assure conformance to acceptable levels.

The design of the daily quality control includes:

- 1. Blanks and appropriate standards are analyzed with each set of samples.
- 2. Ten percent of the samples are run in duplicate for the documentation of precision.
- 3. Ten percent of the samples are spiked to document the recovery and apparent accuracy of the method.
- 4. Where applicable, stable control samples of known content are analyzed with each group of samples.
- 5. Blind samples are submitted to the laboratory without the analysts having knowledge of their origin.

The data gathered from these routine evaluations is used to calculate and maintain both precision (R) and accuracy (P) control limits. These tabulations provide the data rejection criteria for normal laboratory operations. Should the results of replicate or spiked analyses fall outside control limits, the system is thoroughly evaluated, corrective action taken, suspect data rejected and these samples are reanalyzed prior

7. Monitoring Analytical Performance Continued

to reporting. The data tabulations are valuable not only because they provide a current status report on the quality of results but because they are an excellent trouble shooting tool in alerting the laboratory to trends in accuracy and precision of data before out of control situations occur. In addition, they provide documentation of the levels of precision and accuracy at any point in time should the validity of analysis be questioned at a later date.

It is the responsibility of the analysts and Quality Assurance Coordinator to see that prescribed quality control is performed properly and duly recorded.

Recent quality control records for all parameters regularly determined in the laboratory are maintained in designated ring binders and are kept in the office area adjacent to the laboratories (Figure 8).

Older records are in file storage boxed in the record storage room.

CANTON ANALYTICAL LABORATORY PRECISION AND ACCURACY QUALITY CONTROL DATA

PARAMETER	UNITS METHOD	SAMPLE TYPE
CONCENTRATION RANGE.	DETECTION LIMIT	MINIMUM RANGE

Sample		Reagent Method Standard			Precision-Duplicate Analyses				Matrix Effect Spikes				
Date	Number	Analyst	Blank	True Value	Obs. Value	% Recovery	A	В	Diff., R	Mean, (A+B/2)	Spike Conc.	Obs.	% Spike Recover)
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8. Instruments and Calibration

Canton Analytical Laboratory maintains all instruments and major equipment on specified calibration schedules.

Oven and incubator temperatures are checked each day of use. If a trend in inaccuracy is found which cannot be corrected by laboratory personnel, professional service is obtained.

Analytical balances are checked with Class S weights monthly. If inaccuracies are found that cannot be corrected internally, professional services are obtained.

Atomic absorption spectrophotometers are calibrated for each metal analyzed and a record is kept of instrument response. Should a lack of sensitivity or other malfunction be detected that cannot be corrected in-house, professional service is obtained. Service contracts are maintained for these instruments.

8. Instruments and Calibration Continued

UV/visible spectrophotometers are checked with standard solutions before use. If malfunctions cannot be corrected, professional service is obtained.

Gas chromatographs are calibrated before use for sensitivity and accuracy. If the required sensitivity cannot be obtained, prescribed maintenance procedures are initiated. If instrument response is not enhanced to the desired level, professional service is obtained.

9. Chemicals, Solvents and Gases

The purity is specified for all chemical reagents, solvent and gases used in the laboratory. ACS analytical reagent grade chemicals are specified for most test methods.

Standard solutions are stored in borosilicate glass bottles or polyethylene containers, which ever is appropriate. Containers are dated and initialled by the preparer. When prescribed shelf-lives are reached, solutions are discarded and freshly prepared.

All organic and inorganic chemicals are dated upon arrival at the laboratory to monitor shelf life.

Fuels used for atmoic absorption are commercial grade. Nitrogen and Argon carrier gasses are high purity dry grade. Nitrous Oxide Oxidant is anesthetic grade. Air supplied by a compressor passes through a drier and a filter to remove oil, water and trace metals.

The nitrogen, air and hydrogen used for gas chromatography applications are ultra high purity.

The total organic carbon analyzer requires high purity-dry nitrogen.

Ultra high purity carbon dioxide and oxygen gases are used for total organic halide (TOX).

10. Water

There are two grades of reagent water used in the laboratory. The ASTM criteria for reagent water type is shown in Table III and taken from ASTM DII93-77.

The tap water used in the laboratory is from the . Ypsilanti water supply. Its primary use is for the washing of glassware.

The water is checked daily to determine that the proper quality is being maintained. A permanent record of the specific conductance of the deionized water is kept in a control book.

11. Glassware Cleaning Procedures

The cleaning method for glassware is dependent upon the use to which it will be put. General use glassware is washed in a dishwasher with hot water and nonphosphate detergent. It receives a final distilled water rinse.

Detailed washing procedures used for laboratory glassware are given in Appendix B.

12. Personnel

The laboratory is operated by an experienced staff.

The laboratory staff is supplemented by Ph.D. level consultation in quality control, radioactivity, microbiology, industrial hygiene, and limnology. These consultants are available for work on any client's project and supplement the routine work of the laboratory. All staff resumes are available for inspection upon request.

13. Facilities and Expertise

Canton Analytical Laboratory was established in 1977 as an analytical laboratory specializing in environmental analysis of water, wastes, and hazardous substances.

The laboratory contains approximately 6,000 square feet of space and is equipped with modern instrumentation, traditional wet chemistry set-ups, and specialized apparatus.

The laboratory has considerable experience in the analysis of water, wastewater, sediment, air, sewage sludge, industrial wastes, gases, coal, and petroleum products as well as experience in the collection of samples. Canton Analytical Laboratory prides itself in offering these analyses in a timely and cost effective manner. Turn-around time for most analyses is less than one week. Canton Analytical Laboratory uses extensive quality assurance methods designed specifically for each project in addition to an extensive in-house quality assurance program. In addition, Canton Analytical Laboratory participates in the EPA quality assurance programs. Canton Analytical Laboratory is certified by the Michigan Department of Public Health for drinking water analysis and for the analysis of lead in blood. (for coliform, maybe

14. Safety

It is dangerous to assume that personnel at any level of work have adequate information about laboratory safety. Little emphasis is placed on safety or toxicology in college chemistry curriculums. For this reason, Canton Analytical Laboratory has developed a Comprehensive Safety Program (Appendix C).

Each employee is expected to familiarize herself/ himself with the safety rules and regulations and is responsible for safety on the job for himself and fellow workers.

Management is responsible for providing safe operating conditions.

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 Connecticut, USA.
- (10) Federal Register/Vol. 44, No. 233/Mon. Dec. 3, 1979/ Proposed Rules, Part III, EPA, Guidelines Establishing Test Procedures for the Analysis of Pollutants.
- (11) Technicon Instrument Corporation, March 1974, Product Labeling for the Technicon Autoanalyzer II Continuous Flow Instrument, Technicon Instrument Corporation.

 Tarrytown, New York.

TABLES

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TABLE I

Parameter	Method	Detection Limit	Reference
Acidity	Potentiometric	0.01 mg/l as CaCO ₃	Method 305.1-U.S. EPA ¹
Alkalinity	Potentiometric	2 mg/l as CaCO ₃	Method 310.1-U.S. EPA ¹
Bacteria			
Coliform, fecal	Membrane filter	Negat i ve	Page 937-Standard Methods ²
Coliform, total	Membrane filter	Negat i ve	Page 928-Standard Methods ²
Fecal Streptococci	Membrane filter	Negat i ve	Page 944-Standard Methods ²
Standard Plate Count	Agar	Negat i ve	Page 908-Standard Methods ²
Boron	Curcumin, Colorimetric	-0.1 mg/1	Method 212.3-U.S. EPA
Bromide	Titrimetric	2 mg/1	Method 320.1-U.S. EPA
Chloride	Titrimetric, Silver Nitrate	2 mg/l	Page 303-Standard Methods ²
Chlorine	Titrimetric,DPD	0.1 mg/1	Page 332-Standard Methods ²
Cyanide	Titrimetric,Silver Nitrate Colorimetric,Barbituric Acid	0.1 mg/l 0.01 mg/l	Page 317-Standard Methods ⁷
Dissolved Oxygen	Modified Winkler	0.5 mg/l	Method 360.2-U.S. EPA ¹
Fluoride	Distillation followed by SPADNS	0.05 mg/l	Page 393-Standard Methods ²

TABLE I

Parameter	Method	Detection Limit	Reference
Hardness, total	Titrimetric, EDTA	5 mg/l	Method 130.2-U.S. EPA
Hydrogen ion, pH	Electrometric	0.1 unit	Method 150.1-U.S. EPA
l o d i de	Colorimetric	0.1 mg/l	Page 397-Standard Methods ²
Metals Aluminum	Atomic Absorption, flame	0.10 mg/l	Method 202.1-U.S. EPA
Antimony	Atomic Absorption, flame	0.10 mg/l	Method 204.1-U.S. EPA
Arsenic	Atomic Absorption, hydride	0.002 mg/!	Method 206.3-U.S. EPA ¹
Barium	Atomic Absorption, flame	0.10 mg/I	Method 208.1-U.S.EPA
Beryllium	Atomic Absorption, flame	0.01 mg/l	Method 210.1-U.S EPA
Bismuth	Atomic Absorption, flame	0.2 mg/l	Perkin-Elmer Manual ⁹
Cadmium	Atomic Absorption, flame	0.01 mg/1	Method 213.1-U.S. EPA ¹
Calcium	Atomic Asborption, Flame .	0.10 mg/1	Method 215.1-U.S. EPA ¹
Chromium	Atomic Absorption, flame	0.02 mg/l	Method 218.1-U.S. EPA1
Chromium, hexavalent	Colorimetric	0.02 mg/1	Pg. 187-Standard Methods ⁷
Cobalt	Atomic Absorption, Clame	0.01 mg/l	Method 219.1-U.S. EPA
Copper	Atomic Absorption, Clame	0.01 mg/f	Method 220.1-U.S.EPA ¹

TABLE I

Parameter Me	ethod	Detection Limit	Reference
tron At	tomic Absorption,flame	0.02 mg/!	Method 236.1-U.S. EPA ¹
Lead At	tomic Absorption,flame (0.05 mg/I	Method 239.1-U.S. EPA ¹
Lithium At	tomic Absorption,flame	0.01 mg/l	Perkin-Elmer Manual ⁹
Magnesium At	tomic Absorption, flame	0.05 mg/l	Method 242.1-U.S. EPA ¹
Manganese At	tomic Absorption,flame	0.01 mg/l	Method 243.1-U.S. EPA ¹
Mercury At	tomic Absorption, cold vapor	0.0005 mg/l	Method 245.1-U.S. EPA ¹
Molybdenum At	tomic Absorption,flame	0.10 mg/1	Method 246.1-U.S. EPA
Nickel At	tomic Absorption,flame	0.02 mg/l	Method 249.1-U.S. EPA
Potassium At	tomic Absorption,flame	0.02 mg/l	Method 255.1-U.S. EPA
Selenium At	tomic Absorption,hydride	0.001 mg/l	Method 270.3-U.S. EPA
Silicon At	tomic Absorption,flame	0.1 mg/I	Perkin-Elmer Manual ⁹
Silver At	tomic Absorption,flame	0.01 mg/L	Method 272.1-U.S. EPA ¹
Sodium At	comic Absorption, flame	0.1 mg/l	Method 273.1-U.S. EPA
Thallium At	omic Absorption,flame	0.02 mg/1	Method 279.1-U.S. EPA
l'in At	omic Absorption, flame	1.0 mg/1	Method 282.1-U.S. EPA 1
l'itanium At	omic Absorption,flame	0.10 mg/l	Method 283.1-U.S. EPA1

TABLE [

Parameter	Method	Detection Limit	Reference
Vanadium	Atomic Absorption, flame	0.2 mg/l	Pg. 257-Standard Methods ⁷
Zinc	Atomic Absorption, flame	0.01 mg/1	Method 289.1-U.S. EPA ¹
Nitrogen			
Ammonia	Manual Nesslerization Manual Potentiometric	0.01 mg/l 0.05 mg/l	Method 350.2-U.S. EPA Method 350.3-U.S. EPA
Kjeldahl	Colorimetric, Automated Phenate	0.2 mg/I	Method 351.1-U.S. EPA
Nitrate-Nitrite	Cadmium Reduction	0.10 mg/1	Method 353.5-U.S. EPA
Nitrite	Manual Colorimetric	0.005 mg/l	Method 354.1-U.S. EPA ¹
Oxygen Demand BOD (Biochemical Oxygen Demand)	Modified Winkler	1.0 mg/1	Page 543-Standard Methods ²
COD (Chemical Oxygen Demand)	litrimetric	l mg/l	Method 410.1-U.S. EPA ¹
Oil and Grease	Extraction-Gravimetric	0.2 mg/1	Method 413.1-U.S. EPA
Phenols	Spectrophotometric,MBTH with Distillation	0.005 mg/l	Method 420.3-U.S. EPA
Phosphorous Orthophosphate	Colorimetric, Stannous Chloride	0.01 mg/l	Pg. 417-Standard Methods ⁷
fotaf	Persulfate Digestion Colorimetric, Stannous Chloride Red	0.002 mg/1	Page 415 & 417-Standard Methods ⁷

TABLE J

Parameter	Method	Detection Limit	Reference
Residue Total	Gravimetric	l mg/l	Method 160.3-U.S. EPA
Total, Volatile	Gravimetric	l mg/l	Method 160.4-U.S. EPA
Filterable	Gravimetric	l mg/l	Method 160.1-U.S. EPA
Non-filterable	Gravimetric	l mg/l	Method 160.2-U.S. EPA ¹
Specific Conductance	Wheatstone Bridge	0.05 umhos/cm	Method 120.1-U.S. EPA
Sulfate	Turbidimetric	l mg/1	Method 375.4-U.S. EPA
Sulfide	Methylene blue photometric	0.02 mg/1	Pg. 503-Standard Methods ²
Surfactants	Colorimetric, Methylene blue	0.1 mg/l	Method 425.1-U.S. EPA
Tannin & Lignin	Colorimetric	0.1 mg/l as Tanni 0.3 mg/l as Ligni	n Pg. 607-Standard Methods ²
Total Organic Carbon	UV Persulfate Oxidation	2.0 mg/I	Method 415.2-U.S. EPA
Turbidity	Nephelometric	0.1 NTU ·	Method 180.1-U.S. EPA
Organic Compounds Purgeable Halocarbons	Gas Chromatography	varies with level of inte rference	Method 601-U.S. EPA ¹⁰
Purgeable Aromatics	Gas Chromatography	varies with level of interference	Method 602-U.S. EPA ¹⁰
Acrolein/Acrylonitrile	Gas Chromatography	varies with level of interference	Method 603-U.S. EPA 10

ANALYTICAL METHODOLOGY

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Parameter		Method	Detection Limit	Reference
Phenols		Gas Chromatography	varies with level of interference	Method 604-U.S. EPA 10
Phthalate Est	ters	Gas Chromatography	varies with level of interference	Method 606-U.S. EPA ¹⁰
Nitrosamines		Gas Chromatography	varies with level of interference	Method 607-U.S. EPA ¹⁰
Organochtorii Pesticides		Gas Chromatography	varies with level of interference	Method 608-U.S. EPA ¹⁰
Nitroaromatio Isophorone	es and	Gas Chromatography	varies with level of interference	Method 609-U.S. EPA ¹⁰
Polynuclear <i>i</i> Hydrocarboi		Gas Chromatography	varies with level of interference	Method 610-U.S. EPA ¹⁰
Haloethers		Gas Chromatography	varies with level of interference	Method 611-U.S. EPA 10
Chlorinated I	Hydro- carbons	Gas Chromatography	varies with level of interference	Method 612-U.S. EPA ¹⁰
Total Organic I	Halide	Pyrolysis/Microcoulometry	0.005 mg/L	Method 450.1 Interim - U.S. EPA

ANALYTICAL METHODS AND REFERENCES

National Interim Primary Drinking Water Regulations

•	E.P.A	. Referenc	_e (1)
Parameter	Methodology (pa	ge number) Major Equipment
Arsenic	Atomic absorption, hydride	206.3	Atomic absorption spectrophotometer with recorder
Barium	Atomic absorption	208.1	Atomic absorption spectrophotometer with recorder
Cadmium	Atomic absorption	213.1	Atomic absorption spectrophotometer with recorder
Chromium	Atomic absorption	218.1	Atomic absorption spectrophotometer with recorder
Lead	Atomic absorption	239.1	Atomic absorption spectrophotometer with recorder
Mercury	Flameless atomic absorption	245.1	Atomic absorption spectrophotometer with recorder
Nitrate	Cadmium reduction	353.3	Spectrophotometer
Selenium	Atomic absorption, hydride	270.3	Atomic absorption spectrophotometer with recorder
Silver	Atomic absorption	272.1	Atomic absorption spectrophotometer with recorder
Fluoride	Colorimetric, SPADNS	340.1	Spectrophotometer
Chforinated hydrocarbons: Endrin Lindane Methoxychlor Toxaphene	Gas chromatography	(2)	Kuderna-Danish glassware, gas chromatograph equipped with glass-lined injection port and electron-capture detector, and recorder
Chlorophenoxys: 2,4-D 2,4,5-TP	Gas chromatography	(2)	Kuderna-Danish glassware, gas chromatograph equipped with glass-lined injection port and electron-capture detector, and recorder
trihalomethanes	Gas chromatography	(3)	Purge and trap

References

- (1) "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1979.
- (2) "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water, Interim", EPA, EMSL-Cincinnati, June 1979.
- (3) Part to Appendix C of 40 CFR 141 in Federal Register of November 29, 1979 as corrected by Federal Register of March 11, 1980.

TABLE II

SAMPLE PRESERVATION

CANTON ANALYTICAL LABORATORY

<u>Parameter</u>	Minimum Required Sample Volume	U.S. EPA-Recommended ^a Preservation Methods	Container Type ^b	U.S. EPA-Recommended ^C Holding Time
Acidity	100 ml	Cool, 4°C	P,G	14 days
Alkalinity	100 ml	Cool, 4°C	P,G	14 days
Ammonia	400 ml	Cool, 4 ⁰ C H ₂ SO ₄ to pH<2	P,G	28 days
Bacteria Coliform, fecal and total	100 ml	Cool, 4°C 0.008% Na ₂ S ₂ O ₃ f	P,G	6 hours
Fecal streptococci	100 ml	Cool, 4°f 0.008% Na2S203f	* P,G	6 hours
Biochemical oxygen deman	nd 1000 ml	Cool, 4°C	P,G	48 hours
Biochemical oxygen dema Carbonaceous	nd, 1000 ml	Cool, 4°C	P , G	48 hours
Bromide	100 ml	None required	P,G	28 days
Chemical oxygen demand	50 ml	Cool, 4°C H _S SO ₄ to pH ₂	P,G	28 days
Chloride	50 m1	None required	P,G	28 days
Chlorine, total residua	1 200 m1	Determine on site	P,G	2 hours
Color	50 ml	Cool, 4°C	P , G	48 hours
Cyanide, total and amenable to chlorinati	500 ml	Cool, 4 ⁰ C NaOH to pH>12 0.0 g asorbic acid	P,G	14 days

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TABLE II

SAMPLE PRESERVATION

CANTON ANALYTICAL LABORATORY

Parameter	Minimum Required Sample Volume	U.S. EPA-Recommended ^a Preservation Methods	Container Type	U.S. EPA-Recommended ^C Holding Time
Dissolved oxygen Probe	300 ml	Determine on site	G bottle and top	1 hour
•	300 111	poternine on site	o poccie and cop	1 nour
Winkter	300 ml	fix on site	G bottle and top	8 hours
Fluori de	300 ml	None required	P,G	28 days
Hardness	100 ml	HNO ₃ to pH<2	P, G	6 months
pH (Hydrogen ion)	25 ml	Determine on site	P,G	6 hours
Kjildahl and organic nitrogen	500 ml	Cool, 4°C H ₂ SO ₄ to pH<2	Р, G	28 days
Metals		a0a		•
Chromium VI	100 ml	Cool, 4°C	P,G	· 24 hours
Mercury	100 ml	HNO3 to pH<2	P,G	28 days
Metals except above	100 ml	HNO ₃ to plk 2	P,G	6 months
Nitrate	100 mt	Cool, 4°C	P,G	48 hours
Nitrate-nitrite	100 ml	Cool, $4^{\rm O}$ C 11_2 SO $_4^{\rm o}$ to pIK 2	P,G	28 days
Nitrite	50 ml	Cool, 4°C	P,G	48 hours
Oil and Grease	1000 mi	Cool, 4° C H_2 SO $_4$ to pH<2	G	28 days

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TABLE IT

SAMPLE PRESERVATION

Parameter	Minimum Required Sample Volume	U.S. EPA-Recommended ^a Preservation Methods	Container Type b	EPA-Recommended ^C Holding Time
Organic Carbon	100 ml	Cool, 4°C H ₂ SO ₄ to pH<2	P , G	28 days
Organic Halides Total	250 ml	No head space	G, Teflon-lined caps	14 days
Purgeable	40 ml VOA Bottle	No head space	G, Teflon-lined septum	m 14 days
Organic Compounds Extractables (includ phthalates, nitrosam organochlorine pesti PCB's, nitroaromatic polynuclear aromatic haloethers, chlorina and TCOD)	ines cides, s, isopherone, hydrocarbons,	Cool, 4°C 0.008% Na ₂ S ₂ 0 ₃ f	G, Teflon-lined caps	7 days (until extraction) 30 days (after extraction)
Extractables (phenol	s) 1000 ml	Cool, 4° C H_2SO_4 to $pH<2$ 0.008% $Na_2S_2O_3$	G, Teflon-lined caps	7 days (until extraction) 30 days (after extraction)
Purgeables (halocarb and acromatics)	ons (2) 40 ml VOA Bottles	Cool, 4°C 0.008% Na ₂ S ₂ 0 ₃ No head space ³	G, Teflon-lined septu	m 14 days
Purgeables (Acrolein and Acrylonitrile)	(2) 40 ml VOA Bottles	Cool, 4°C 0.008%Na ₂ S ₂ 0 No head Space	G, Teflon-lined septu	m 3 days
Orthophosphate	50 ml	Filter on site Cool, 4°C	P,G	48 hours

TABLE II

SAMPLE PRESERVATION

Parameter	Minimum Required Sample Volume	U.S. EPA-Recommended ^a Preservation Methods	Container Type	EPA-Recommended ^C Holding Time
Pesticides	1000 ml	Cool, 4°C 0.008%Na ₂ S ₂ 0 ₃ f	G, Teflon-lined cap	7 days (until extraction) 30 days (after extraction)
Phenols	500 ml	Cool, 4°C H ₂ SO ₄ to pH<2	G	28 days
Phosphorous, elemental	50 ml	Cool, 4°C	G	48 days
Phosphorous, total	50 ml	Cool, 4°C H ₂ SO ₄ to pH<2	P,G	28 days
Radiological Alpha, Beta and Radio	ım 1000 ml	None .	P,G	6 months
Residue, total	- 100 ml	Cool, 4°C	P,G	14 days
Redidue, filterable	100 ml	Cool, 4°C	P,G	14 days
Residue, nonfilterable	100 ml	Cool, 4°C	P,G	7 days
Residue, settleable	1000 ml	Cool, 4°C	P,G	7 days
Residue, volatile	100 ml	Cool, 4°C	P,G	7 days
Silica	50 ml	Cool, 4°C	P	28 days
Specific conductance	100 ml	Cool, 4°C	P,G	28 days
Sulfate	50 ml	Cool, 4°C	P,G	28 days

TABLE II

SAMPLE PRESERVATION

CANTON ANALYTICAL LABORATORY

Parameter	Minimum Required Sample Volume	U.S. EPA-Recommended ^a Preservation Methods	Container Type	U.S. EPA-Recommended ^C Holding Time
Sulfide	500 ml	Cool, 4°C Zinc acetate-NaOH to	P,G pH>9	7 days
Sulfite	50 ml	None	P,G	Immediately
Surfactants	250 ml	Cool, 4°C	P., G	48 hours
Temperatur e	1000 ml	Determine on site	Р, G	Immediately
Turbidity	100 ml	Cool, 4°C	P,G	48 hours

- a. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- b. Polyethylene (P) or Glass (G).
- c. Samples should be analyzed as soon as possible after collection. The times listed are maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee or the laboratory has data on file to show that the specific types of samples under study are stable for the longer time.

Some samples may not be stable for the maximum time period given in the table. A permittee, or the laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

- d. Samples should be filtered immediately on-site before adding preservatives for dissolved metals.
- e. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific organic compounds.
- f. Should only be used in the presence of residual chlorine.

TABLE II

SAMPLE PRESERVATION

NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS - Sample collecting, handling, and preservation.

Parameter	Preservative	Container	Holding Time
Arsenic*	2.0 ml 1:1 HNO ₃	500 ml plastic	6 months
Barium*	"	H	•
Cadmium×	H .	H	**
Chromium*	H	11	Ħ
Lead*	•	11	н
Selenium*	**	#	**
Silver*	Ħ	Ħ	••
Mercury*	н	H	14 days
Nitrate	1.0 ml 1:1 H ₂ SO ₄	250 ml plastic	14 days
Fluoride	None	250 ml plasttic	1 month
Chlorinated hydrocarbons	Refrigerate at 4°C	l liter amber glass** (2 bottles per sample)	14 days***
Chlorophenoxys	Refrigerate at 4°C	l liter amble glass** (2 bottles per sample)	7 days***
Trihalomethanes		25 ml glass, teflon- lined septa, (2 bottles per sample)	14 days

All parameters analyzed from one 500 ml plastic bottle

Foil-lined cap used.

Well-stoppered and refrigerated extracts may be held up to 30 days.

TABLE III
CRITERIA FOR REAGENT WATER

	Type I	Type II	Type III	Type IV
Total matter, max mg/l	0:1	0.1	1.0	2.0
Electrical Conductivity, max, mho/ cm at 298 K (25 C)	0.06	1.0	1.0	5.0
Electrical Resistivity, min M -cm at 298 K (25 C)	16.67	1.0	1.0	0.2
pH at 298 K (25 C)	(A) -	· (A)	6.2 to 7.5	5.0 to 8.0
Minimum color retention time of potassium permanganate, minutes	60	60	10	10
Maximum soluble silica	NOT DETE	CTABLE	10 g/l	No limit

Microbiological classification (B)

- (A) The measurement of pH in Type I and II reagent waters is meaningless and has been eliminated from the procedure, since electrodes used in this test contaminate the water.
- (B) When bacterial levels need to be controlled, reagent grade types should be further classified as follows:

•	Type A	ľype B	Type C
Maximum total bacterial count	0/m1	10/ml	100/ml

TABLE IV

REAGENT SHELF LIFE

<u>Parameter</u>	Reagent	Container Type	Storage or Shelf Life	Comments
Acidity	$\mathrm{H_2O_2}$ (30% solin.	glass or plastic	indefinite	store in refrigerator
	NaOH (0.02N)	plastic	3 months	discard if turbid
	H_2SO_4 (0.02N)	glass or plastic	6 months	restandardize monthly
Alkalinity	Na_2CO_3 (0.05N)	glass or plastic	3 months	
	H ₂ SO ₄ (0.1N)	glass or plastic	6 months	,
	H_2SO_4 (0.02N)	glass or plastic	6 months	restandardize monthly
,	Indicator solution 1	glass or plastic	indefinite	
	Indicator solution 2	glass or plastic	indefinite	
Bacteria Coliform, fecal and total	Phosphate buffer sol'n.	glass or plastic	3 months	store at 4°C, discard if turbid
	MF-C Broth	glass	l week	refrigerate
	M Endo Broth	glass	. I week	refrigerate
Boron	Stock boron sol'n.	plastic	3 months	
	Curcumin reagent	plastic	I week	store at 4°C

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
Bromide	Acetic acid sol'n.	glass	l week	
	Potassium Bromide sol'n.	amber glass	indefinite	
	Indicator solution	glass or plastic	indefinite	
·	Chbramine-T solution	amber glass	1 month	refrigerate
Chloride	K2CrO4	glass or plastic	indefinite	
	AgNO ₃ (0.0282N)	amber glass	6 months	restandardize monthly,
	NaCl (0.0282N)	glass or plastic	indefinite	store out of light
Chlorine	Potassium Iodide Crystal	amber glass	indefinite	
	Phosphate buffer sol'n.	glass or plastic	6 months	discard if colored or if
	FAS Titrant	glass or plastic	1 month	mold is present
	DPD indicator sol'n.	amber glass	indefinite.	discard when discolored
Cyanide, total and amenable to chlorination	NaOH (IN)	ptastic	indefinite	discard is sediment forms
	Ascorbic acid	glass	indefinite	
	Magnesium chloride	glass or plastic	indefinite	
	Ca(OC1) ₂ (5%)	amber glass	1 month	store in dark
	$\mathbf{H_2So_4}$	glass or plastic	indefinite	

TABLE IV

REAGENT SHELF LIFE

CANTON ANALYTICAL LABORATORY

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
(Titrimetric)	Indicator solution	glass or plastic	indefinite	keep tightly capped
	AgNO ₃ (0.0192N)	amber glass	6 months	restandardize monthly, store in the dark
(Colorimetric)	Chloramine-T	amber glass	1 week	store in refrigerator
	Cyanide stock sol'n.	glass or plastic	3 months	check titer each week
	Cyanide standard sol'n.	glass or plastic	1 day	
	Phosphate buffer	glass or plastic	6 months	store in refrigerator
	Pyridine-barbituric acid	glass or plastic	6 months	store in the dark
Fluoride	Naf stock sol'n.	plastic	indefinite	•
. •	SPADNS sol'n.	glass or plastic	indefinite	store in refrigerator
	Zirconyl-acid reagent	glass or plastic	indefinite	
	Acid zirconyt-SPADNS	glass or plastic	l year	store in refrigerator
	Sodium arsenite sol'n.	glass or plastic	indefinite	
Hardness	Buffer sol'n.	plastic	1 month	
•	EDTA (0.01M)	plastic	indefinite	restandardize monthly
	Indicator solution	plastic	indefinite	
	Standard Ca solution	glass or plastic	indefinite	,

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REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life Comments
Hydrogen ion (pH)	Buffer solution .	glass or plastic	follow manufacturer's recommendations
lodide	Stock indide solution	glass	indefinite
	Citric buffer solution	glass or plastic	indefinite check pll before use
	Indicator solution	amber glass	6 months
	NH ₄ OH (2M)	glass or plastic	6 months discard if sediment forms
	Potassium peroxy- monosulfate solution	glass or plastic	6 months
	Sodium thiosulfate sol'n.	glass or plastic	6 months
Metals	Various salt solutions	glass or plastic	not to ex- follow suppliers ceed I year recommendations
Nitrogen Ammonia (manual)	Nessler reagent	amber glass	l year
Nesslerization	Borate buffer	glass or plastic	indefinite
	Boric acid solution	glass or plastić	indefinite
	'Stock ammonia	glass or plastic	indefinite
	Standard ammonia	glass or plastic	3 months
Ammonia (manual) Potentriometric	NaOH (10N)	glass or plastic	l year

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
Ammonia _.	Stock ammonia	glass or plastic	indefinite	
•	Standard ammonia	glass or plastic	3 months	•
Kjeldahl	н ₂ 'S0 ₄ (4%)	glass or plastic	indefinite	
	Digestion solution	glass	indefinite	
	NaOH (20%)	plastic	indefinite	
	Stock buffer solution	glass	indefinite	
	Sodium nitroprusside (0.03%)	glass or plastic	indefinite	
	Sodium hypochloride (6%)	glass or plastic	l week	
	Stock standard	glass or plastic	indefinite	
	Standard solution	glass or plastic	3 months	
Nitrate-Nitrite (Cadmium Reduc-				
tion)	NED-Di hydroch Lor i de	amber glass .	l month	
	Sulfanilamide	glass or plastic	3 months	
	EDTA-NH4C1 Solution	plastic	1 month	discard if darkened
	Cadmium metal	glass	indet in ite	•

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
Nitrate-Nitrite	HC1	glass or plastic	indefinite	
	CuSO ₄	glass or plastic	indefinite	
	Stock nitrate	glass or plastic	indefinite	
	Standard nitrate	glass or plastic	prepare fresh	1
Nitrite	Sulfanilamide	glass or plastic	3 months	
	NED -Dihydrochloride	amber glass	1 month	
	HC1 (1+2)	glass or plastic	indefinite	
	Stock nitrite	glass or plastic	3 months	preserve with 1 ml of chloroform
	Standard nitrite	glass or plastic	prepare fresh	
Oxygen Demand BOD	Phsophate buffer	glass or plastic	3 months	store in refrigerator, discard if turbid
	$MgSO_{4}$ solution	glass or plastic	indefinite	
	CaCl ₂ solution	glass or plastic	indefinite	
	ferric chloride sol'n.	glass or plastic	indefinite	•
	Acid and alkali sol ¹ n.	glass or plastic	indefinite	•
	Sodium Thiosulfate sol'n.	glass	2 months	

TABLE 1V

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
COD	K ₂ Cr ₂ O ₇ (0.0250N)	glass or plastic	indefinite	
	H ₂ SO ₄ reagent	glass or plastic	indefinite	
	Fe(NH ₄) ₂ (SO ₄) ₂ .6H ₂ 0	glass or plastic	indefinite	standardize before use
	Ferroin indicator	glass or plastic	in de finite	
Oil and Grease	HC1	glass or plastic	indefinite	
	Freon	glass or plastic	in de finite	
Phenots	CuSO4 5H20	glass or plastic	indefinite	
	4-Aminoantipyrene	glass or plastic	make fresh	
	Distilation Reagent(10%)	glass or plastic	indefinite	
	Buffer solution	glass or plastic	3 months'	discard if turbld or darkened
	Potassium Ferric Cyanide Solution	glass or plastic	i week	
	Chloroform	glass	indefinite	
	Stock phenol	glass or plastic	. 6 months	discard if darkened
·	Standard phenot	glass or plastic	make fresh	discard if darkened
Phosphorous	$H_2 so_4$ (5N)	glass or plastic	indefinite	
	PhonolphthaleIn	glass or plastic	indefinite	

TABLE IV

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
Phosphorous (continued)	Ammonium molybdate	plastic .	indefinite	store in refrigerator
	Stannous chloride	glass or plastic	indefinite	
	Stock phosphorous sol'n.	glass or plastic	indefinite	
	Standard phosphorous solution	glass or plastic	in de finite	
Specific conductance	KC1 (0.0100M)	pyrex	indefinite	·
Sulfate	Conditioning reagent	plastic or glass	indefinite	
	BaCl ₂ crystals	plastic or glass	indefinite	•
	H ₂ SO ₄ solutions	plastic or glass	indefinite	
Sulfide	Amine-sulfuric acid stock solution	amber glass	indefinite	
	Amine-sulfuric acid standard solution	brown glass	3 months	discard if colored
	FeC 1 3 6 H 2 0	glass or plastic	indefinite	
	(NH ₄) ₂ HPO ₄	glass or plastic	indefinite	
•	Methylene blue solin. 1	glass or plastic	indefinite	
	Methylene blue sol'n. 11	glass or plastic	indefinite	

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Storage or Shelf Life	Comments
Surfactants	Stock LAS	glass	indefinite	biodegradeable; store in refrigerator
	Standard LAS	glass	prepare fres	
	Chloroform	glass	indefinite	
	NaOH (IN)	plastic	indefinite	discard if turbid
	H ₂ SO ₄	glass or plastic	indefinite	
	Methylene blue sol'n.	glass or plastic	indefinite	
	Wash solution	glass or plastic	indefinite	
Tannin & Lignin	Tannin-lingin reagent	glass or plastic	indefinite	absence of green color
	Carbonate-tartrate	glass or plastic	indefinite	store in refrigerator
·	Stock solution	glass or plastic	indefinite	
	Standard solution	glass or plastic	indefinite ·	
Total Organic Carbon	Potassium persulfate (IM)	glass or plastic	in defini te '	: :
	HNO ₃ concentrate	glass or plastic	indefinite	
Turbidity	Stock formazin suspension	glass or plastic	t month	

REAGENT SHELF LIFE

Parameter	Reagent	Container Type	Shelf Life	Comments
Turbidity (Continued)	Standard formazin suspension	glass or plastic	1 week	
Organic Compounds	Stock and standard solutions	glass	vartous	store in refrigerator

TABLE V
Reporting of Results

Microbiological Parameters

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Biochemical Oxygen Demand (5 days)	1.0 m/g/l	1.0 - 10 10 - 100 100 - 1000 1000 - 10000	x.x xx xxo xxoo xxoo
Coliform	0 count/100 ml	0 - 10	X XX
Standard Plate Cour	nt 0	0 - 10 10 - 100	X XX

TABLE V

	ROUTINE		SIGNIFICANT
PARAMETER	DETECTION LIMITS	RANGE	FIGURES -
Aluminum (flame)	0.10 mg/l	0.10 - 1.0	0.XX
		1.0 - 10	X . X
		10 - 100	XX
*		100 - 1000	xxo
Antimony (flame)	0.10 mg/l	0.10 - 1.0	o.xx ·
•		1.0 - 10	x.x ·
		10 - 100	xx
		100 - 1000	xxo
Arsenic (hydride)	0.002 mg/1	0.002 - 0.010	0.00x
mcl=0.05	op.	0.010 - 0.10	0.0XX
•	25% gmcl	0.10 - 1.0	0.XX
	·	1.0 - 10	x . x
Barium (flame)	0.10 mg/1 /2 10%	0.10 - 1.0	0.XX
MCL = 10	o te	1.0 - 10	$X \cdot X$
•		10 - 100	XX
		100 - 1000	XXO
Beryllium (flame)	0.01 mg/1	0.01-0.10	o.ox
		0.10 - 1.0	0.XX
		1.0 - 10	x . x
		10 - 100	XX
		100 - 1000	XXO

Table V
Reporting of Results

Metals

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Cadmium (flame)	0.01 mg/1 No -	0.01 - 0.10	0.0x
mcle 0.010	= mcL	0.10 - 1.0	0 · XX
		1.0 - 10	x • x .
		10 - 100	xx
		100 - 1000	xxo
		•	
Calcium (flame)	0.10 mg/l	0.10 - 1.0	0.XX
		1.0 - 10	$X \cdot X$
		. 10 - 100	XX
		100 - 1000	xxo
Cobalt (flame)	0.01 mg/1	0.01 - 0.10	o.ox
		0.10 - 1.0	o.xx
		1.0 - 10	x . x
·	•	10 - 100	XX
		100 - 1000	xxo
Chromium (flame) 0.02 mg/l	0.02 - 0.10	o.ox
	407 17 MCL	0.10 - 1.0	o.xx
1102 - 0.05	10/0	0.10 - 1.0 1.0 - 10	x . x
•	. 4	10 - 100	XX
	·	100 - 1000	XXO

_ 、

TABLE V

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Copper (flame)	. 0.01 mg/l	0.01 - 0.10	0.0X
		0.10 - 1.0	0.XX
	·	1.0 - 10	x . x
		10 - 100	XX
•		100 - 1000	xxo
Iron (flame)	0.02 mg/l	0.02 - 0.10	o.ox
		0.10 - 1.0	0 - XX
		1.0 - 10	X • X
		10 - 100	XX
		100 - 1000	XXO
Lead (flame)	0.05 mg/l	0.05 - 0.10	0.0X
mc1= 0.05	No = mcl	0.10 - 1.0	0.XX
•	= mcl	1.0 - 10	X . X
		10 - 100	XX
		100 - 1000	77.0
Lithium (flame)	0.01 mg/l	0.01 - 0.10	0.0x
		0.10 - 1.0	0. XX
		1.0 - 10	X • X
		10 - 100	XX
		100 - 1000	XXO

TABLE V

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Magnesium (flame)	0.05 mg/1	0.05 - 0.10	o.ox
		0.10 - 1.0	o.xx
		1.0 - 10	x . x
		10 - 100	XX .
		100 - 1000	xxo
Manganese (flame)	0.01 mg/l	0.01 - 0.10	o.ox
		0.10 - 1.0	0. XX
		0.10 - 1.0	0.XX
		1.0 - 10	x . x
		10 - 100	XX
		100 - 1000	XXO
Molybdenum (flame)	0.10 mg/[0.10 - 1.0	o.xx
		1.0 - 10	X • X
	,	10 - 100	XX
		100 - 1000	xxo
Nickel (flame)	0.02 mg/[0.02 - 0.10	o.ox
		0.10 - 1.0	0.XX
		1.0 - 10	X.X
		10 - 100	XX
		100 - 1000	XXO

TABLE V

{ *	PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANI FIGURES
п	Potassium (flame)	0.02 mg/1	0.02 - 0.10	0.0X
			0.10 - 1.0	0.XX
			1.0 - 10	x . x
			10 - 100	XX
	\smile		100 - 1000	xxo
E	_	·		•
L	Selenium (hydride)	0.001 mg/1	0.001 - 0.010	0.00X
ſ	mcl= 0.01	0 f C	0.010 - 0.10	0.0XX
		109. Jmcl	0.10 - 1.0	0 • XX
			1.0 - 10	X - X
	Silica (flame)	0.1 mg/l	0.1 - 1.0	· 0.X
E			1.0 - 10	X . X
L	·		10 - 100	XX
F	en en en en en en en en en en en en en e		100 - 1000	XXO
L				
	Silver (flame)	0.01 mg/1	0.01 - 0.10	o.ox
t	mll= 0.05	0.01 mg/ op 20%, 7 mcL	0.10 - 1.0	0.XX
		20%, 5/11	1.0 - 10	X • X
E		, -	10 - 100	XX
-			100 - 1000	XXO
•		•	·	
:	Titanium (flame)	0.10 mg/l	0.10 - 1.0	0.X
-		•	1.0 - 10	X • X
A			10 - 100	XX

TABLE V
Reporting of Results

PARAMETER DE	ROUTINE ETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Zinc (flame)	0.01 mg/1	0.01 - 0.10	0.0x
		0.10 - 1.0	0.XX
		1.0 - 10	x . x
		10 - 100	xx -
		100 - 1000	XXO
Mercury (cold vapor)	0.0005 mg/l	0.0005 - 0.001	0.000X
Mercury (cord vapor)	_	.0010010	0.00XX
mc1 0.002	ok 25% J Mci	.010 - 0.10	.033
	25% 7 MCC	0.10 - 1.0	• XX
	U .	1.0 - 10	X
	•	1.0 - 10	X • X
Bismuth (flame)	.2 mg/1	.2 - 1.0	. X
		1.0 - 10	x . x
		10 - 100	XX
Sodium (flame)	0.1 mg/1	0.1 - 1.0	0.8
		1.0 - 10	X.X
		10 - 100	XX
		100 - 1000	XXO
Thalium (flame)	0.02 mg/l	0.02 - 0.10	0.0X
		0.10 - 1.0	0 . XX
		1.0 - 10	N. X
		10 - 100	XX
tin (flame)	1.0 mg/l	1.0 - 10	X.X
		10 - 100	XX
		100 - 1000	XXO

TABLE V
Reporting of Results
Conventional Parameters

	PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
•	Cyanide, Total	0.1 mg/1	0.1 - 1.0	0 . X
	(potentiometric)		1.0 - 10	x . x
L			10 - 100	ХX
r			100 - 1000	xxo ·
L			•	
C:	Cyanide, Total	0.01 mg/1	0.01 - 0.10	0.0X
	(colorimetric)		.0.10 - 1.0	0.XX
~			1.0 - 10	X . X
			10 - 100	$\mathbf{X}\mathbf{X}$
•			. 100 - 1000	XXO
0				
U	Iodide	0.1 mg/l	0.1 - 1.0	0. X
ſ:			1.0 - 10	X • X
C	(10 - 100	XX
r			100 - 1000	XXO
			1000 - 10000	XX00
r	Alkalinity	2.0 mg/1	2.0 - 10	x - x
L			10 - 100	xx
r			100 - 1000	xxx
			1000 - 10000	XXOO
•	Hexavalent Chromiu	m 0.02 mg/l	0.02 - 0.10	0.0x
1			0.10 - 1.0	0.XX
•			1.0 - 10	X • X
٠.			10 - 100	XX
			100 - 1000	XXO

TABLE V

Reporting of Results

PARAMETER D	ROUTINE ETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Methylene Blue Active Substances	0.1 mg/1 LAS	0.1 - 1.0	0 Y
Active Substances	O.1 mg/1 LAS	1.0 - 10	0.X
			X • X
		10 - 100	X X
Phenolics	0.005 mg/1	0.005 - 0.010	0.00x
		0.010 - 0.10	o.oxx
·		0.10 - 1.0	o.xx
		1.0 - 10	٧.٨
		10 - 100	XX
		100 - 1000	XXO
Bromide	2.0 mg/l	2.0 - 10	X . X
		10 - 100	XX
•		100 - 1000	XXO
	•	1000 - 10000	XXOO
Chlorine, Residual	0.1 mg/1	0.1 - 1.0	0.x
		1.0 - 10	X • X
		10 - 100	XX
	•	100 - 1000	xxo
fluoride	0.05 mg/l	0.05 - 0.10	0.0x
	<u>.</u>	0.10 - 1.0	0.XX
		1.0 - 10	X • X
		10 - 100	XX
		100 - 1000	XXO

TABLE V
Reporting of Results

	PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
-	Boron	0.1 mg/1	0.1 - 1.0	0. X
(i			1.0 - 10	x . x
			10 - 100	XX
			100 - 1000	XX0 -
r: _	Chemical Oxygen	. /-		
	Demand	1 mg/1	1 - 10	X
_			10 - 100	XX
			100 - 1000	$\chi \chi \alpha$
			1000 - 10000	XXOO
	Formaldehyde	0.3 mg/l	0.3 - 1.0	0 • X .
_			1.0 - 10	X - X
0	•		10 - 100	XX
			100 - 1000	XXO
•	Hardness	5 mg/l	5 - 10	X
Γ			10 - 100	ХX
			100 - 1000	XXO
r			1000 - 10000	xxoo
	Sulfate	1 mg/l	1 - 10	<u>,</u> X
7			10 - 100	XX .
1			100 - 1000	XXO
•		•	1000 - 10000	XX00

TABLE V
Reporting of Results

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Sulfide	0.02 mg/l*	0.02 - 0.10	0.0X
		0.10 - 1.0	O. XX
		1.0 - 10	x . x
		10 - 100	xx ·
		100 - 1000	xxo
Total Organic Carbo	n 2.0 mg/l	2.0 - 10	x . x
		10 - 100	XX
		100 - 1000	XXO
		1000 - 10000	xxoo
Acidity	0.01 mg/1	0.01 - 0.10	o.ox
·	·	0.10 - 1.0	O.XX
		1.0 - 10	X . X
		10 - 100	XX
		100 - 1000	xxo
рН	0.1 S.U.	0.1 - 1.0	0.x
•	•	1.0 - 10	x . x
	•	10 - 14	xx.x
Specific Conductanc	e 0.05 umhos/cm	0.05 - 0.10	o.ox
		0.10 - 1.0	0.XX
		1.0 - 10	x . x
		10 - 100	XX
		100 - 1000	XXX
		1000 - 10000	XXXO

^{*} Sample volume dependent

TABLE V
Reporting of Results

PARAMETER	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANI FIGURES
Turbidity	O.1 NTU	0.1 - 1.0	0.X
		1.0 - 10	x . x
•		10 - 100	ХX
		100 - 1000	xxo .
		1000 - 10000	XXOO
Total Organic			
Halide	.005 mg/1	0.005 - 0.010	0.00X
		0.010 - 0.10	0.088
		0.10 - 1	$O \cdot XX$
		1 - 10	X • X
		10 - 100	XX
		100 - 1000	XXO
		1000 - 10000	XXOO

TABLE V
Reporting of Results

Nutrients

PARAMETER	ROUTINE DETECTION LIMIT	RANGE	SIGNIFICANT FIGURES
Nitrate	0.10 mg/1	0.10 - 1.0	o.xx
mcl = 10	υ/ -	1.0 - 10	x . x
	1% 7 mcL	10 - 100	ХX
		100 - 1000	xxo -
Chloride	2 mg/l	2 - 10	x _
		10 - 100	XX
		100 - 1000	XXO
		1000 - 10000	XXOO
Nitrite-N	0.005 mg/l	0.005 - 0.010	0.00x
		0.010 - 0.10	o.oxx
	·	0.10 - 1.0	o.xx
		1.0 - 10	X • X
		10 - 100	XX
		100 - 1000	XXO
Kjeldahl Nitrogen	0.20 mg/l	0.20 - 1.0	.
	3 , 1	1.0 - 10	0. XX
		10 - 100	X . X
		100 - 1000	XX XXO
Total Phosphorus	0.002 mg/L	0.002 - 0.010	o.oox
		0.010 - 0.10	o.oxx
		0.10 - 1.0	0. XX
•		1.0 - 10	X.X
		10 - 100	XX

TABLE V
Reporting of Results

Nutrients

PARAMETERS	ROUTINE DETECTION LIMITS	RANGE	SIGNIFICANT FIGURES
Ortho Phosphorus	0.01 mg/1	0.01 - 0.10	0.0x
		0.10 - 1.0	0.XX
		1.0 - 10	x. x
		10 - 100	XX
	•	100 - 1000	XXO
Ammonia-N	0.02 mg/1	0.02 - 0.10	o.ox
(automated-phenate)		0.10 - 1.0	0.XX
		1.0 - 10	$X \cdot X$
		. 10 - 100	XX
		100 - 1000	XXO
Ammonia-N	0.04 mg/l	0.04 - 0.10	0.0X
(manual potentiomet	etric)	0.10 - 1.0	0.XX
		1.0 - 10	$X \cdot X$
•		10 - 100	XX
		100 - 1000	XXO

TABLE V
Reporting of Results

Residue

PARAMETERS	ROUTINE DETECTION LIMIT	RANGE	SIGNIFICANI FIGURES
Oil and Grease	0.5 mg/l*	0.5 - 1.0	O. X
		1.0 - 10	x . x
	•	10 - 100	XX
	,	100 - 1000	xxo
		1000 - 10000	xx00
Residue, Total	1 mg/l	1 - 10	x
Total, Volatile		10 - 100	XX
Filterable		100 - 10000	$\lambda \lambda 0$
Non-Filterable		1000 - 10000	XX00
,		10000 - 100000	XX000

, .,

^{*} sample volume dependent

APPENDICES

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CHAIN-OF-CUSTODY PROTOCOL

Appendix A Canton Analytical Laboratory Chain-of-Custody Protocol Quality Assurance Manual

SAMPLE CONTROL

A sample* is physical evidence collected from a facility or from the environment. An essential part of this investigations effort is that the evidence gathered be controlled. To accomplish this, the following sample identification and chain-of-custody procedures have been established.

SAMPLE IDENTIFICATION

The method of identification of a sample depends on the type of measurement performed. An in-situ measurement is one in which the sample is collected and/or the measurement performed and the data are recorded directly in logbooks or field Data Records (FDR's). with identifying information while in the possession of the sampling team. Examples of in-situ measurements are pH, temperature, conductivity, flow measurement, continuous air monitoring, and stack gas analysis.

Samples other than in-situ measurements are identified by a sample tag (page 75) or other appropriate identification (hereinafter referred to as a sample tag) attached to or folded around the sample. Included on the tag are the sample identification number, date, time and location of sample collection, designation of the sample as a grab or composite, the type of sample and preservation, any remarks, and the signature of the sampler. Each sample's identification number consists of a three-digit project code assigned by the Laboratory Project Leader and listed in the project plan, and a two-digit (three as required) sequence number assigned by the sampler(s) at the time of sample collection. At each station, the sequence number begins at 01 for each sample type and is increased by one (1) each time a sample is collected or composited for the particular sample type, regardless of date or time. Thus, the example, if the 10th "metals" sample was collected at the same time as the 17th "nutrients" sample

^{*} For purposes of this manual, the term 'sample' includes remote sensing imagery.

CAL Q

CANTON ANALYTICAL LABORATORY

Environmental Analysis
153 Elder Street / Ypsilanti, MI 48197 (313) 483-7430

Leb No.	Date		Time	
Client Name			Comp.	Grab
Sample Point				
Parameters		Preserv.		
	<u>-</u>			
Sampler: (Signature)				
Remarks:				
		·		

at the same station, the sequence numbers would be 10 to 17, respectively. The information listed above is also recorded in the appropriate logbook along with any pertinent in-situ measurement data and field observations. Thus, while provisions will be made where circumstances require a slight modification to the number of sequence for a specific survey, a typical sample number will be:

Project Code

Station No.

Sequence No.

202

03

07 or, 202-03-07

After collection and identification, the sample is preserved and maintained under the chain-of-custody procedures discussed below. If the composite or grab sample collected is to be split with another company, it should be aliquated into similar sample containers. Sample tags with identical information are attached to each of the samples, with the Company tag being marked as "Company Split". The same procedure is followed when splitting samples with Federal or State agencies; the appropriate agency is marked on the split tag. In a similar fashion, all tags on blank or duplicate samples will be marked "Blank" and "Duplicate", respectively.

CHAIN-OF-CUSTODY PROCEDURES

Due to the evidentiary nature of samples collected during enforcement investigations, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample possession, chain-of-custody procedures are followed.

Sample Custody

A sample is under custody if:

- 1. It is in your actual possession, or
- 2. It is in your view, after being in your physical possession, or
- 3. It was in your physical possession and then you locked it up to prevent tampering, or
- 4. It is in a designated secure area.

Field Custody Procedures

- In collecting samples for evidence, collect only that number which provides a fair representation of the media being sampled. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible should handle samples.
- 2. The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or properly dispatched.
- 3. Sample tags shall be completed for each sample, using waterproof ink unless prohibited by weather conditions.
- 4. During the course and at the end of the field work, the Laboratory Project Leader determines whether these procedures have been followed, and if additional samples are required.

Transfer of Custody and Shipment

- 1. Samples are accompanied by a Chain-of-Custody Record. When transferring the possession os samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents transfer of custody of samples from the sampler to another person, or to an analytical laboratory.
- 2. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate Record prepared for each laboratory. Shipping containers will be padlocked for shipment to the laboratory. The "Courier to Airport" space on the Chain-of-Custody Record shall be dated and signed.

- 3. Whenever samples are split with a facility or government agency, a separate Chain-of-Custody Record is prepared for those samples and marked to indicate with whom the samples are being split.
- 4. All packages will be accompanied by the Chain-of-Custody Record showing identification of the contents. The original Record will accompany the shipment.
- 5. If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Laboratory Bill of Lading should be used. Receipts from post office and bills of lading will be returned as part of the permanent documentation.

Laboratory Custody Procedures

- 1. A sample custodian or a designated alternate will receive samples for the laboratory and verify that the information on the sample tags matches that on the Chain-of-Custody Record included with the shipment. The custodian signs the custody record in the appropriate space; a laboratory staff member performs this function in the field. Couriers picking up samples at the airport, post office, etc., shall sign in the appropriate space.
- 2. The custodian distributes samples to the appropriate analysts.

 The names of individuals who receive samples are recorded in internal Laboratory records. Laboratory personnel are responsible for the care and custody of samples from the time they receive them until they return them to the custodian.
- 3. Once field sample testing and necessary quality assurance checks have been completed, the unused portion of the sample is kept in a securred area for a minimum of 30 days.

 All identifying tags, data sheets and laboratory records shall be retained as part of the permanent documentation.

DOCUMENT CONTROL

The goal of the Document Control Program is to assure that all documents for a specific project issued to the Contractor, Canton Analytical Laboratory, will be accountable when the project is completed. This program includes a serialized document number system, a document inventory procedure, and a central filing system, all under the supervision of Canton Analytical Laboratory.

Accountable documents used by Laboratory employees shall include items such as logbooks, field data records, correspondence, sample tage, graphs, chain-of-custody records, bench cards and photos (see page #86 for a more complete list). Each document bears a serialized number and is listed with the number, in a project document inventory assembled at the project's completion.

Unless prohibited by weather, waterproof ink is used in recording all data on serialized accountable documents.

SERIALIZED DOCUMENTS

The Laboratory is responsible for assigning the necessary serialized documents to project personnel for field activities.

Once a Leader is appointed, all field logbooks, field data records, field laboratory logbooks, sample tags and chain-of-custody records are assigned to this person. The Leader is responsible for ensuring that a sufficient supply of documents is obtained for an investigation and that these documents are properly distributed to the appropriate personnel. This provides the Leader with a list of all serialized project documents that were assigned to personnel for field activities.

PROJECT LOGBOOKS

The logbook of the Leader will document the transfer of logbooks to the individuals who have been designated to perform specific tasks on the survey. All pertinent information should be recorded in these logbooks from the time each individual is assigned to the project until the project is completed.

Logbook entries should be dated, legible and contain accurate and inclusive documentation of an individual's project activities. Since the logbook forms the basis for the later written reports, it must contain only facts and observations. Language should be objective, factual and free of personal feeling or other terminology which might prove inappropriate. Entries made by individuals other than the person to whom the logbook was assigned are dated and signed by the individual making the entry.

Field analysts who conduct their assigned project analyses in a mobile laboratory are assigned a team logbook by the Leader. In addition to information documenting the analysis performed, field analysts document in their logbooks the data and results of any calibration of mobile laboratory equipment. A record is also kept of any incidents related to the survey; for example, the electricity going off in the lab, tampering with project vehicles or equipment, etc. When appropriate, visitors to the mobile lab, such as facility personnel, are noted in the logbook.

All project logbooks are the property of Canton Analytical Laboratory to be turned over to the Leader when a survey assignment has been concluded.

FIELD DATA RECORDS

Where appropriate, serialized Field Data Records (in the form of individual sheets or bound logbooks) are maintained for each survey sampling station or location. The Leader numbers the FDR's with the appropriate project code and station number. All in-situ measurements and field observations are recorded in the FDR's with all pertinent information necessary to explain and reconstruct sampling operations. Each page of a Field Data Record is dated and signed by all individuals making entries on that page. The Leader and the field team on duty are responsible for ensuring that FDR's are present during all monitoring activities and are stored safely to avoid possible tampering.

SAMPLE IDENTIFICATION DOCUMENTS

Assignments of all serialized sample tags to field personnel is recorded in the Leader's logbook. Individuals are accountable for each tag assigned to them until it has been filled out, attached to a sample, and transferred to another individual with the corresponding Chain-of-Custody Record. At no time are any sample tags to be discarded; if any of these forms are lost, voided or damaged, it is noted in the appropriate FDR or logbook immediately upon discovery. Tags attached to those samples split with a facility will be accounted for as described below.

At the completion of any reconnaissance or field-sampling investigation, all unused sample tags are to be returned to the Leader by the individual to whom they were originally assigned. This individual lists the serial numbers of the returned items in the Leader's logbook and signs and dates the transfer.

CHAIN-OF-CUSTODY RECORDS

All serialized Chain-of-Custody Records are assigned and accounted for in a manner similar to that for the sample tags as described above. When samples are transferred from a field sampler or courier to field laboratory personnel, the analyst, after signing, retains the original custody record and files it in a safe place. The copy of the custody record is returned to the Leader. A similar procedure is followed when dispatching samples via common carrier, mail, etc., except that the original accompanies the shipment and is signed and retained by the receiving laboratory sample custodian.

When samples are split with the facility or another government agency, the separate custody record that is prepared (see page #77) is labeled to indicate this. In addition, the serial numbers from all the tags are recorded on the custody record. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the sample. If a representative is unavailable or refuses to sign, this is noted in the "received by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time. The copy of the custody record may be given to the facility or agency upon request; all originals are returned to the Leader.

All laboratory observation and calculations not recorded on serialized bench cards, instrument graph printouts, etc., are entered in serialized logbooks assigned by a file custodian. The logbook should contain information sufficient to recall and describe succintly each step of the analysis performed should the analyst be required to testify in subsequent enforcement proceedings. Sufficient detail should be provided to enable others to reconstruct the analysis should the analyst not be available to do so. Any irregularities observed during the testing process should be noted. If, in the technical judgement of the analyst, it is necessary to deviate from a particular analytical method, the deviation shall be properly justified and documented.

When an individual is assigned a logbook for use on a variety of projects, each page contains information about only one project and is labeled with the project code, dated, and signed by the individual. All bench cards, instrument printouts, and other separate documents are labeled similarly. Notes (taken at meetings, from re-

search articles, etc.) which do not relate to a particular project shall not be kept in the assigned logbook. When a laboratory logbook is completed, it is returned to the file custodian and a new logbook is issued. The custodian or other appropriate staff member maintains an inventory sheet for the logbook, listing the project code for each page. These books that have been completed and turned in are used for reference purposes only.

where applicable, the file custodian issues a serialized instrument logbook in which all information relating to calibration and maintenance of a particular laboratory instrument is recorded. A serialized sample entry logbook is used in the laboratory to record the entry of the samples to the laboratory or laboratory instrument for analysis. Again, each page should contain information about one project only.

PHOTOGRAPHS

When movies, slides or photographs are taken which visually show the effluent or emission source and/or any monitoring locations. they are numbered to correspond to logbook entries. The name of the photographer, date, time, site location, and site description are entered sequentially in the logbook as photos are taken. Once develope—the slides or photographs should be serially numbered corresponding to the logbook descriptions.

CORRECTIONS TO DOCUMENTATION

As previously noted, unless prohibited by weather conditions, all documentation in logbooks, FDR's, sample tags, custody records and other data sheets are filled out with waterproof ink. None of the accountable serialized documents listed above are to be destroyed or thrown away even if they are illegible or contain inaccuracies which required a replacement document.

If an error is made in a project logbook assigned to one individual, that individual may make corrections simply by crossing a line through the error and entering the correct information. Changes made subsequently are dated and initialed. If an error is discovered on a sample tag, custody record, or FDR, when possible the person who made the error should correct it. Corrections or insertions are made by inserting the word or abbreviation for "corrected", the date, and the correcting person's initials beside the correction. The procedure applies to words or figures inserted or added to a prior recorded statement.

If a sample tag is lost in shipment, or a tag was never prepared for a sample(s), or a properly tagged sample was not transferred with a formal Chain-of-Custody Record, the following procedure applies. A written statement is prepared detailing how the sample was collected, air-dispatched or hand-transferred to the field or laboratory. The statement should include all pertinent information, such as entries in field logbooks regarding the sample, whether the sample was in the sample collector's physical possession or in a locked compartment until hand-transferred to the laboratory, etc.

Copies of the statement are distributed to the Leader and the appropriate project files.

CONSISTENCY OF DOCUMENTATION

Before releasing any analytical sample results to the Leader, the Field Analysts and/or Laboratory Analysts assemble and cross-check information on corresponding sample tags, custody records, bench cards, analyst logbooks and sample entry logbooks to ensure that data pertaining to each particular sample is consistent throughout the record. A statement that all project evidentiary data in the Laboratory's possession has been accounted for accompanies the transfer on any analytical data from the Analyst to the Leader.

The leader then conducts a cross-check of evidentiary data in his possession (FDR's, logbooks, custody records, etc.) to ensure that information recorded corresponds to information from each of the Analysts and is consistent throughout the project record.

DOCUMENT NUMBERING SYSTEM AND INVENTORY PROCEDURE

In order to provide document accountability to the appropriate individuals, each of the document categories discussed above features a unique serialized number for each item within the category. Logbooks, FDR's, sample tags and custody records are serially numbered before assignment to project personnel. The logbooks and FDR's are usually given a five-digit number, with the project code as the first three digits followed by a two digit document number. Sample tags and custody records are labeled with a four digit document number and the project code appears elsewhere on the document. All documentation not covered by the above (logbooks, data sheets, graphs, etc.) are uniquly and serially numbered using the project code as part of the number when appropriate.

All other documents (such as recorder graph paper, data calculation sheets, memos, correspondence, photos, etc.) which are generated during a project are sequentially numbered with the project code, initialed, and a serialized number, usually at the time the file is assembled.

FILES

After a Leader has completed his work for a particular investigation, all documents generated from that project should be assembled in the file. Individuals may retain clean (no handwritten comments) copies of documents for their personal files but only after personally verifying that the original or similar copy is in the file.

The Leader is responsible for assuring the collection, assembly, and inventory of all documents relative to a particular project at the time the project objectives are completed. The file then becomes accountable. Any records leaving the file must be signed out.

PROJECT FILE

When the Laboratory has completed the project objectives, all inventoried file documents are reviewed. By this time each document will have been labeled with a unique serialized number as specified above. The format of the File covers the following document classes.

- A. Project Logbooks
- B. Field Data Records
- C. Sample Identification Documents
- D. Chain-of-Custody Records
- E. Analytical Logbooks, Log Data, Calculations, Bench Cards, Graphs, etc.
- F. Correspondence
 - 1. Intra-office
 - 2. EPA
 - 3. Industry
 - 4. Record of Confidential Material
- G. Report Notes, Calculations, etc.: Drafts
- H. References, Literature
- I. Sample (on-hand) Inventory
- J. Check-out Logs
- K. Litigation Documents
- L. Miscellaneous photos, maps, drawings, etc.

Once deposited in the File, documents may only be checked out through Canton Analytical Laboratory or designated representative.

REPORTS

All draft reports are numbered and accountable. The author is responsible for disseminating draft reports for internal review, and preparing a memorandum for the Leader to transmit copies to the Client.

All draft copies of the report are to be returned to the Leader. Once comments have been incorporated and the final report has been prepared, all draft copies are disposed of.

LITIGATION DOCUMENTS

Any court documents, litigation reports, letters, memos, etc., from the EPA, State Pollution Control offices, etc.; which discuss legal matters or strategies, should be placed in a separate file folder which is reviewed by Laboratory personnel at the appropriate time.

CONFIDENTIAL INFORMATION

Any information received with a request of confidentiality is handled as "confidential". A separate, locked file is maintained by the Chain-of-Custody custodian for the segregation and storage of all confidential and trade-secret information. Upon receipt, this information is directed to and recorded in the Confidential Inventory Log. The information is then made available to authorized personnel, but only after it has been logged out. The information shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by and under the supervision of Canton Analytical Laboratory. reproduction should be kept to an absolute minimum. All copies will be entered into the document control system and the same requirements will apply as for the original. In addition, this information may not be entered into any computer or data handling system. documents may not be destroyed except upon approval by and under the supervision of Canton Analytical Laboratory. The Company shall remove and retain the cover page of any confidential information disposed of shall be removed and retained for one year and a record of the destruction shall be kept in the Confidential Inventory Log.

GLASSWARE CLEANING PROCEDURES

Appendix B
Glassware Cleaning Procedures
Quality Assurance Manual

GLASSWARE CLEANING PROCEDURES

A. All field sample bottles are new and not washed. From each new lot of incoming sample bottles, a randomly selected bottle is filled with deionized water, let set 24 hours, and all parameters are analyzed to determine if contamination exists.

B. General Laboratory Glassware

- Remove any foreign material in glassware or container with phosphate-free laboratory detergent solution, hot water and brush.
- 2. Rinse thoroughly with warm water to remove all detergent.
- 3. Rinse thoroughly with deionized water.
- 4. If a grease film remains, use acctone to dissolve and remove the grease. Then follow general washing procedures (Steps 1, 2, and 3).
- 5. If any additional residue remains, rinse and/or soak the item with 1+1 hydrochloric acid (HCl) or chromic acid cleaning solution, then follow the general washing procedure (Steps 1, 2, and 3).

C. BOD Bottles and Dilution Water Bottles for Bacteria Analysis:

- 1. Empty bottles and retain caps to be washed by hand.
- Rinse bottles with warm tap water and place upside down in dishwasher.

- 3. Put appropriate amount of dishwasher detergent (phosphate-free) in dishwasher.
- 4. Operate dishwasher through full cycle.
- 5. Wash bottle caps by "general laboratory glassware" procedure (B).
- 6. Remove bottles from washer and place bottles and caps in the appropriate cabinet in the laboratory.

D. Metals Glassware

- 1. Remove any debris in the glassware or container with detergent, tap water, and a brush.
- If residue is still present, soak in warm chromic acid cleaning solution.
- 3. Rinse the glassware or container with 1+1 nitric acid (HNO3).
- Rinse with demineralized-distilled water three successive times.

E. Odor Glassware

- Wash with phosphate-free laboratory detergent solution and hot water.
- 2. Rinse thoroughly twice with warm tap water.
- 3. Rinse thoroughly with sulfuric acic cleaning solution
- 4. Rinse thoroughly with demineralized water.
- 5. Use immediately.

F. Automatic ISCO Sampler Bottles

- 1. Empty the bottles, rinse with tap water and brush out excess debris.
- 2. Operate dishwasher through full cycle.
- 3. Remove the bottles from the dishwasher after drying and place in the field department storage area.
- 4. Before being taken into the field, bottles are acid rinsed with 1+1 nitric acid.
- 5. Rinsed three times with deionized water.
- G. Spectrophotometer Cells (Curvettes)
 - 1. DO NOT USE BRUSHES TO CLEAN CELLS.
 - 2. Rinse with 1+1 nitric acid NOT sulfuric acid cleaning solution.
 - 3. Rinse with demineralized water at least three times.

NOTE: Handle curvettes from top (open end) only.

Be careful to avoid contact between surface of curvettes and other hard surfaces. DO NOT leave fingerprints or spots on curvettes.

- H. Special Procedures for Removing Substances from Heated Glassware
 - 1. Remove any debris from the glassware with phosphate-free laboratory detergent solution and a brush.
 - 2. Rinse thoroughly with tap water.

- 3. Soak the glassware in a chromic acid cleaning solution for at least 15 minutes. If debris does not come off, continue to soak in the acid solution for a longer period of time, possibly overnight.
- 4. Rinse with tap water.
- 5. Rinse with demineralized water three times minimum.

I. Oil Flasks

- 1. Clean as in general glassware and container procedures (B).
- Fill flask with chromic acid cleaning solution for a relatively long period of time, preferably overnight.

J. Gas Chromatography Glassware

- Wash the glassware in hot water with phosphate-free laboratory detergent.
- Rinse with hop tap water.
- If any trace of residue remains, soak the glassware in chromic acid cleaning solution for at least 15 minutes.
- 4. Rinse with hot tap water to flush away loosened material and until washings are acid free.
- 5. Rinse with acetone.
- Air dry glassware and store in designated drawers, in organic area.

 Flush the glassware just before using with the same solvent to be used in the analysis.

K. Pesticide and Herbicide Sample Containers

- New 1 liter glass bottles with caps are rinsed with pesticide grade methylene chloride.
- 2. Bottles and caps are oven dried in an inverted position.
- 3. Bottles are cooled, capped, and shipped to the job-site.

CANTON ANALYTICAL LABORATORY SAFETY PROCEDURES

Appendix C Safety Procedures Quality Assurance Manual

SAFETY PROCEDURES

1. Laboratory Conduct:

Follow instructions exactly.

Perform only authorized experiments.

Protect eyes, face, hands, and body.

Practice good housekeeping.

Learn basic first aid.

Know where to get help quickly.

Know location of first aid and fire fighting equipment.

Report all accidents and unusual occurrences immediately.

Be professional.

2. Personal Safety:

Safety glasses are required.

Clean up all water spills on the floor.

Use only equipment and tools suited to the job at hand.

Dispose of broken glass only in the marked container.

Do not wear loose clothing or open-top shoes. Wear a laboratory coat, a rubber apron, goggles, or gloves when indicated.

Avoid working along in the laboratory.

3. Fire Prevention

Whenever possible, and always when large quantities are involved, use flammable liquids in a fume hood. When it is necessary to use flammables on an open bench, be certain that there are no flames nearby.

Place waste flammable liquids in the appropriate safety cans for disposal.

Dispose of solid and liquid oxidants, such as peroxides, perchlorates, and nitrates, by flushing down the sink with water. Keep these materials away from flammable items such as wood and paper.

Be certain not to overload electrical circuits.

Do not use equipment with worn or bare wiring.

Be aware of the two types of fire extinguishers in the laboratories. The dry chemical (2A40BC) type is for use on paper, liquid, or electrical fires. The CO2 (BC) type if for use on liquid, or electrical fires.

Know the location of the fire extinguishers, blankets, and fire alarms.

4. Prevention of Poisoning

Use toxic materials such as chlorine gas, cyanides, and bromine in a hood only. These are inhalation hazards, and some are toxic by skin absorption.

Use gloves when handling bromine.

Some compounds in use in the lab are slow acting poisons when injested or absorbed in small amounts. Among these are arsenic, mercury, lead, and hexavalent chromium compounds. Wear gloves when handling these compounds in high concentrations and wash hands thoroughly after use.

Clean up all chemical spills, even of seemingly harmless materials. One spill may react with another. Neutralize concentrated acids with sodium carbonate (Na2CO3), and bases with boric acid (H3BO4) before cleaning up.

Always use a rubber bulb to pipet.

Exercise care in handling of all samples. Their contents are unknown.

Do not eat, drink, or smoke in the lab work areas.

If you have any questions about handling a particular compound or reaction, consult your supervisor, or the (RC Handbook of Laboratory Safety.

Safety Equipment

Know how to use the following items in the Laboratory: Fire extinguishers, fire blanket, fire alarm. safety showers, eye wash stations, and first aid kits.

6. General

Protective Clothing - Each employee will be provided a sufficient number of laboratory garments (i.e., lab coats, smocks, etc.) to be worn at all times while in the laboratory. Each employee should have one clean garment available at all times.

Eye Protection - Safety glasses are required under Federal law and must be utilized. Custom-fit safety glasses or corrective lenses are furnished by Canton Analytical Laboratory.

Foot Protection - Conventional street-type footwear is sufficient. Sandals, canvas, or similar footwear should not be worn in the laboratory.

<u>Miscellaneous</u> - Rubber, cloth, or leather gloves are available for hand protection and must be worn whenever the occasion warrants.

Exits - Be conscious at all times of the nearest laboratory exit and nearest building exit.

Smoking - Smoking is strictly prohibited in the laboratory.

Food - Do not eat or drink in the laboratory or store lunches in refrigerators used for chemicals.

SOIL

PARAMETER	(REFERENCE) METHOD	(REFERENCE) METHOD		
Asbestos	(2) P & CAM 239	(3)		
Aluminum	(1) 202.1	(1) 202.1		
Chromium	(1) 218.1	(1) 218.1		
Barium	(1) 208.1	(1) 208.1		
Beryllium	(1) 210.1	(1) 210.1		
Cobalt	(1) 219.1	(1) 219.1		
Copper	(1) 220.1	(1) 220.1		
Iron	(1) 236.1	(1) 236.1		
Nickel	(1) 249.1	(1) 249.1		
Manganese	(1) 243.1	(1) 243.1		
Zinc	(1) 289.1	(1) 289.1		
Boron	(1) 212.3	(1) 212.3		
Vanadium	(1) 286.1	(1) 286.1		
Silver	(1) 272.1	(1) 272.1		
Arsenic	(1) 206.3	(1) 206.3		
Antimony	(1) 204.1	(1) 204.1		
Selenium	(1) 270.3	(1) 270.3		
Thallium	(1) 279.1	(1) 279.1		
Mercury	(1) 245.1	(1) 245.1		
Tin	(1) 282.1	(1) 282.1		
Cadmium	(1) 213.1	(1) 213.1		
Lead	(1) 239.1	(1) 239.1		
Ammonia	(1) 350.3	(1) 350.3		
Cyanide	(1) 335.3	(1) 335.3		
Sulfide	(1) 376.2	(1) 376.2		
Acid Extractables	(4) 604	(5) 8040		
Base/Neutrals	(4) 625	(5) 8090,8100,8110,8120		
Volatiles	(4) 601, 602	(5) 8010,8020,8030		
Pesticides	(4) 608	(5) 8080		
PCB	(4) 608	(5) 8080		
Dioxins	(4) 613	(5) 8130		
Thiram	(4) 608	(5) 8080		

References:

- -(1) Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March 1979.
- (2) NIOSH Manual of Analytical Methods, Second edition, Volume 1, U.S. Dept. of Health, Education and Welfare.
- (3) Interim Method for the Determination of Asbestos in Bulk Insulation samples, EPA 600/M4-82-020, December 1982.
- (4) Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.
- (5) Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods, SW 846.